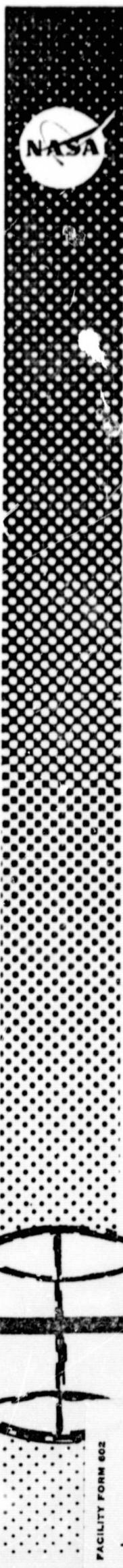


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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA PROGRAM APOLLO WORKING PAPER NO. 1337

NONFLAMMABLE FLUOREL COMPOUNDS

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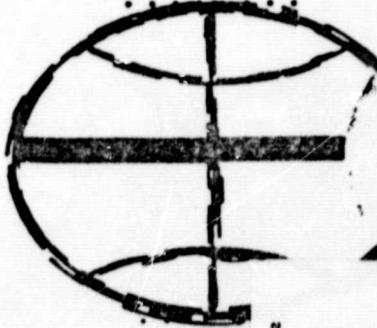
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NASA PROGRAM APOLLO WORKING PAPER NO. 1337

NONFLAMMABLE FLUOREL COMPOUNDS

PREPARED BY

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
MANNED SPACECRAFT CENTER
HOUSTON, TEXAS
April 22, 1968

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As is true of any such group effort, other personnel and companies have contributed their time and assistance. We are grateful for this help and for the additional support which will be required to complete our objectives.

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NONFLAMMABLE FLUOREL COMPOUNDS

By D. E. Supkis

SUMMARY

This program was designed to initiate research on new elastomers and to investigate and develop Fluorel for use as a nonflammable spacecraft material. The Fluorel copolymer, with the addition of fire-retardant ingredients, can be made nonflammable under the most rigid spacecraft conditions of 16.5 psia, bottom ignition, in a 100-percent oxygen atmosphere.

On proper balance of formulary ingredients and technique, the Fluorel mill stock can be foamed, molded, or extruded. By Solvation, the material can be controlled as paste, coating, or spray solution. Such a variety of properties and forms presents exciting possibilities for replacing flammable spacecraft components.

This paper discusses the properties of the more promising Fluorels presently under consideration. These Fluorels are being investigated for a variety of applications and the current effort in each of these categories is presented. Since this is a continuing project, the materials and application information contained in this report are of an interim nature and are intended for periodic revision.

INTRODUCTION

To alleviate the fire hazard which exists in the 100-percent oxygen environment of the Apollo spacecraft, new materials must be developed to replace highly flammable components. In those areas where replacement or accessibility is impracticable, the use of nonflammable barriers to flame propagation will restrain the fire to a minimal hazard area.

Any new nonflammable material developed to replace nonmetal components in the command or lunar modules must necessarily meet all specifications for end use application, in addition to satisfying nonflammabilities. The ideal raw material should then be capable of assuming different forms for a variety of applications. It must exhibit properties comparable to

the component which it will replace, or compatible with the component which it will shield. If foam or sheet, the new material must lend itself to fabrication, as it must prove economical in every phase of manufacturing, design, and application.

Analysis of potential applications for such a new material yields the need for substitutes for highly flammable rubber products, such as boot soles, belts, wire insulation, tubing, and hoses. Adhesive applications include replacement for the epoxy cements used to laminate the spacecraft components and substrates. It would be highly desirable to substitute a nonflammable elastomer for the polyurethane used in helmet liners, crew couches, eyepieces, and equipment packaging.

There are new applications for conformable coatings, caulking compounds, and coatings for space suits. Other nonflammable applications include magnets, fabric edgelock, and electrical harness boots. And there is a need for a material which will provide nonflammability, while improving the physical characteristics of such essential components as fabrics, harnesses, and bladder materials. Finally, a substitute must be developed for the flammable plastics used in the spacecraft.

To satisfy the requirements of these applications, a nonflammable elastomer, in mill sheet or stock form, must be capable of foaming, extrusion, compression molding, and casting. When dissolved in the appropriate solvents, the material must maintain the viscosity necessary for conformable coating and caulking operations, while further dilution would yield a solution which can be spray coated. Further, it should release the solvent under realistic drying conditions.

It would be advantageous if the nonflammable elastomer could be capable of varying degrees of cure to control or alter the physical and chemical characteristics. For additional adaptability, the elastomer should be compatible with a range of other materials which may be added to fulfill the specific requirements of a particular end use.

NONFLAMMABLE MATERIALS DEVELOPMENT

Preliminary Investigation

To reduce the fire hazard in the Apollo spacecraft, a twofold in-house program was initiated:

1. To coat existing irreplaceable and flammable spacecraft components with a fireproofing barrier material

2. To develop entirely new materials which can be fabricated to replace existing spacecraft components

An ideal material would fulfill both objectives, and be capable of a broad range of fabrication properties. A material for use in the spacecraft must satisfy the following nonflammability criteria:

In 100 percent oxygen — at both 16.5 and 6.2 psia
"Materials shall be self-extinguishing when ignited
at the bottom of the test specimen."

This specification is described in Category A of Procedures and Requirements for the Evaluation of Spacecraft Metallic Materials, MSC-A-D-66-3, Revision A, June 5, 1967. This procedures manual also establishes requirements for odor, flash and fire points, total organics outgassing, and nontoxicity.

Early investigation of nylon, coated with a mixture of various inorganic fire retardants, extinguishers, and quenching agents, confirmed the premise that spacecraft components can be made nonflammable under these rigid requirements. This is recognized as a purely laboratory effort, however, as the coating had little adhesion and the fabric lost flexibility.

To increase the flexibility and adhesion, this philosophy was extended by adding these same materials to a latex base, again with the idea of coating materials to make them nonflammable. Polyvinyl acetate (PVA) was first evaluated in this study because of its superior coating properties and adhesion.

Intumescent materials, which produce carbon dioxide, and on heating expand to form a nonflammable, insulating barrier, were added to PVA with some degree of success. Other compounds, which release their waters of hydration when heated, were added to quench ignition and render the PVA nonflammable.

Mixtures of these and other chemicals such as sodium aluminate, calcium silicate, potassium chloride, calcium chloride, sodium carbonate, and sodium borate were successful in retarding fire in a pure oxygen atmosphere.

There was, however, a very basic problem in this approach. Polyvinyl acetate is itself so flammable that the addition of fire retardants in sufficient quantities destroyed those very qualities which had led to its selection.

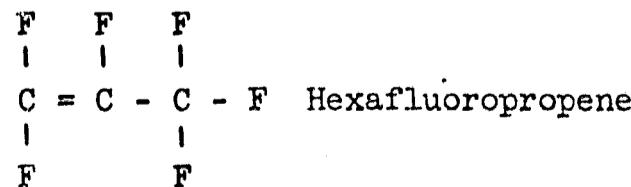
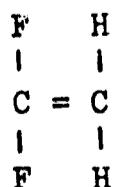
To take the investigation a step further, other flexible bonding materials were needed to achieve the original objectives. This selection itself created problems since the addition of emulsifiers to achieve compatibility between organic and inorganic components increases flammability properties. The same contradiction was true with the addition of plasticizers, drying oils, stabilizers and other formulating ingredients generally used to enhance physical properties. The necessity for placing the elastomer in solution to achieve more versatility adds the extremely flammable solvent to the mixture.

At this point the use of high-halogen-, low-hydrogen-containing elastomers seemed the most likely prospects for achieving the two objectives. These saturated halogens offer little or no available carbon and hydrogen for reaction with the oxygen of the spacecraft atmosphere; thus, they are extremely fire resistant. Since the more active halogens are the most stable, both chlorine- and fluorine-based hydrocarbons were considered.

Polyvinyl chloride (PVC) and chlorinated PVC were some of the chlorine compounds investigated and considered flammable under the rigid requirements of the Category A specification, as the chlorine bond breaks under these conditions, leaving the carbon free for reaction with the oxygen atmosphere. Fluorine-based hydrocarbons showed good flammability properties and represented a logical choice for compounding. However, since this could not be researched in-house, a number of rubber and elastomer suppliers were contacted for sample materials containing fire retardants, extinguishers, and quenching agents to fulfill the objectives of the test program.

Conception of Fluorel Program

Several suppliers were willing to undertake the task of modifying their products to obtain an elastomer tailored to meet Apollo specifications and requirements. The Chemical Division of Minnesota Mining and Manufacturing Company (3M Company), working with saturated and partially saturated fluorine compounds, submitted samples of Fluorel (a commercial elastomer) for evaluation. Fluorel is a copolymer of hexafluoropropene and vinylidene fluoride.



Early samples exhibited very good physical adaptability but burned under spacecraft conditions. However, one formulation, sent for routine analysis, was a "reluctant combustible," evidencing self-extinguishing traits, although it did burn under spacecraft testing conditions.

In an attempt to strike a balance between the need for physical properties and the development of a nonflammable material, several principles of nonflammable behavior were evaluated by 3M Company as additives to the basic Fluorel compound.

Further extrapolation of this formulation yielded more samples, until the greater majority were nonflammable. Physical properties and adaptability showed excellent promise. At this point, the Fluorel test program was conceived to adapt this material for use as a nonflammable material and fireproof coating.

DEVELOPMENT OF NONFLAMMABLE FLUOREL ELASTOMERS

Basic Raw Material Supplier

The 3M Company is the only basic raw material supplier for the special nonflammable Fluorel compounded for and evaluated in this program. The new nonflammable Fluorel compound which was developed has the designation L-2231 and exhibits the following physical specifications. Standard test procedures or American Society of Testing Materials (ASTM) test methods were used to develop this physical data:

Press cure 20 min at 320° F

Oven cure 24 hr at 400° F

Original properties

Tensile strength, psi	2045
Elongation, percent	210
Durometer, Shore A	71
Modulus, 100 percent	705

Air-oven aged 7 days at 400° F

Tensile strength, psi	1720
Elongation, percent	160
Durometer, Shore A	74
Modulus, 100 percent	980
180° bend	Pass

Air-oven aged 2 days at 550° F

Tensile strength, psi	705
Elongation, percent	25
Durometer, Shore A	88
180° bend	Fail

ASTM tear strength (Die C), lb/in.	122
--	-----

Bashore resilience, percent	6
---------------------------------------	---

Compression set, percent

After 22 hr at 400° F	55
---------------------------------	----

Low temperature (Gehman flexibility), °F

T2	+23
T5	+12
T10	+7
T100	-4

Low temperature (Brittle point), °F

0.060 in. thick	-13
---------------------------	-----

Electrical Properties

Volume resistivity, ohm/cm	1.8×10^{13}
Dielectric strength, V/mil	180

Frequency

Dielectric constant at 25° C

100 Hz	18.7
1 kc	17.8
10 kc	16.4
100 kc	13.7

Dissipation factor at 25° C

100 Hz	3.8
1 kc	4.7
10 kc	9.0
100 kc	15

Radiation resistance, 10 Mrad, gamma

Tensile strength, psi	1885
Elongation, percent	160
Durometer, Shore A	77
Modulus, 100 percent	705

The 3M Company has also developed another nonflammable Fluorel designated L-2248 to obtain increased Shore A hardness for a variety of applications. This material has the following physical properties:

Original properties

Tensile strength, psi	1960
Elongation, percent	70
Durometer, Shore A	83 to 85

Air-oven aged 7 days at 400° F

Tensile strength, psi	2065
Elongation, percent	60
Modulus, 100 percent	91

ASTM tear strength (Die C), lb/in.

74

The 3M Company development section is continuing the search for other nonflammable materials based on the requirements of this program.

Under a special arrangement, the 3M Company has divulged the formula information for their L-2231 elastomer to Raybestos-Manhattan Company of North Charleston, South Carolina (also referred to in this paper as Raybestos-Manhattan), and the Mosites Rubber Company of Fort Worth, Texas (also referred to in this paper as Mosites).

To avoid competition with these customers, but to protect their own proprietary interests, this arrangement is exclusive. However, the 3M Company has reserved the right to supply the L-2231 mill stock to other companies for fabrication testing purposes only. This would not include information on the particular ingredients, but the 3M Company would assist with technological support of the other company's effort.

As referred to in this report, the flammable copolymer is differentiated from the nonflammable formula. The formula is known as mill stock when designed for further fabrication. There are only two sources of supply for the mill stock, Raybestos-Manhattan and the Mosites.

Mosites and Raybestos-Manhattan Development Efforts

Mosites 1059 mill stock is the same material which has been received from the 3M Company as their L-2231 formula or which has been prepared by Mosites from the 3M Company formula. Using this material, Mosites has been particularly successful in foaming the mill stock for fabrication into a number of nonflammable spacecraft components.

In addition, they have developed sheet stock from the 1059 formulation which has the following physical properties:

Durometer, Shore A	70
Tensile strength, psi	2063
Elongation, percent	200
Tear strength, psi	108
Compression set, percent	
After 22 hr at 350° F	29.3
Specific gravity	2.02

This material requires postcuring at 400° F for 24 hours for nonflammability. A cinnamon odor is characteristic of the uncured material. When fully cured, it meets specifications as a nonflammable material. This formulation has been successfully fabricated into hoses, electrical harness boots, tubing, magnets, and oxygen masks (fig. 1). When used at a minimum thickness of 50 mils, the 1059 formulation has been successfully used as a fireproof coating on flammable silicone rubber products.

Mosites has also developed new materials using the basic 3M Company formulation which can be used for wire coating and caulking compounds. Another formulation has been successfully evaluated as a nonflammable adhesive.

Raybestos-Manhattan has applied their own designation, L-3217-1, to the 3M Company formula. This compound has the following physical properties:

Press cure	30 min at 320° F
Postcure	24 hr at 400° F

Original properties

Tensile strength, psi	1637
Elongation, percent	187
Durometer, Shore A	72

To achieve nonflammability, L-3217-1 must be postcured at 400° F for 2½ hours. At 16.5 psia, the ignition source destroys sheet thicknesses less than 0.050 inch. For this reason, its use is not recommended at less than that thickness for fireproof coatings. Above this thickness, the L-3217-1 sheet has been successfully fabricated into hoses, tubing, electrical harness boots, and magnets. Tubing of varying diameters is shown in figure 2.

However, Raybestos-Manhattan working primarily with the 3M Company basic Fluorel copolymer, have added their own ingredients for nonflammability properties. A number of Raybestos-Manhattan formulations have been evaluated for impregnating fabrics used in the spacecraft, for wire coating, potting compounds, tubing, hoses, belts, harnesses, and a myriad of other nonflammable spacecraft applications.

Raybestos-Manhattan has also been successful in developing an in-house screening technique which has reduced the number of new materials which must be tested for nonflammability in the Manned Spacecraft Center (MSC) laboratories. This test can accurately determine whether or not a particular Fluorel formula will pass the Category A flammability test.

To test a particular Fluorel formula, ignition is attempted using a propane torch in the presence of a perchlorate stick. The perchlorate stick decomposes on heating to produce oxygen, which simulates the Category A testing procedure.

The perchlorate sticks are inexpensive and can be purchased by contacting Mr. William Moran, Director of Propulsion Marketing, Northrop Carolina, Inc., Asheville, North Carolina.

Testing Procedures and Quality Control

The nonflammability characteristics of the Fluorel elastomer are less sensitive to small variations in the basic copolymer than to changes resulting from improper formulation and mixing. Thus, in addition to quality control procedures inherent in good manufacturing practice or to in-house testing of new products, all batches of elastomer produced for use in this program must be evaluated by the Manned Spacecraft Center for nonflammability under spacecraft conditions.

The 3M Company has agreed to maintain an MSC-approved inventory of the basic copolymer which may be supplied for development and fabrication efforts.

The 3M Company has supplied mill stock to various companies interested in developing new applications for the Fluorel elastomer. At the time the sample was supplied, an identical sample was submitted to MSC for testing. Should the 3M Company sample be nonflammable and the fabricated article made from the sample burn, responsibility for the alteration in flammability characteristics can be ascribed.

In addition, any new formulation by Mosites or Raybestos-Manhattan is sent to the MSC laboratories for flammability evaluation. Arrangements can be made to obtain these compounds from the two companies for development and evaluation.

An Apollo certification program has been initiated to approve promising Fluorel elastomer compounds for spacecraft applications. To assure nonpartisan results, the samples for testing are sent to the White Sands Testing Facility, White Sands, New Mexico. Quality-assured results of Fluorels submitted to date are contained in table I. Only Apollo certified materials will be used on the spacecraft.

ELASTOMERS UNDER INVESTIGATION BASED ON FLUOREL DEVELOPMENT

Mosites Nonflammable Materials

Mosites 1062 Fluorel.- This material is a closed-cell soft sponge exhibiting the following physical properties:

Density, lb/cu ft 15 to 25

Compression-deflection, lb 3 to 5

Compression set, percent

After 24 hr at 75° F 21.5

After 22 hr at 158° F 100

Temperature range -65° to +450° F

Surface Skin both sides

At 6.2 psia in 100 percent oxygen, the 1062 formulation is nonflammable at any thickness. At 16.5 psia, the material burns at a rate of 0.006 to 0.07 in/sec for thicknesses below 3/8 inch.

However, it is the skin resulting from the foaming process that burns fastest (0.157 in/sec) at this pressure. Skinless samples, exhibiting the same physical properties as those with skin, are self-extinguishing above a thickness of 3/8 inch at 16.5 psia.

Samples of the 1062 soft sponge, both with skin and skinless, are illustrated in figure 3. These sponges have been seriously evaluated for fabrication into eyepieces, seat cushions, shock absorbers, and instrument casings.

Mosites 1062-C Fluorel.- This designation refers to a firm sponge sheet with these physical properties:

Density, lb/cu ft	15 to 25
Compression-deflection, lb	7 to 9
Compression set, percent	
After 24 hr at 75° F	24.4
After 22 hr at 158° F	100
Temperature range	-65° to +450° F
Surface	Skin both sides

Both the 1062 and 1062-C sponges are formed from the basic Mosites 1059 formula. The 1062-C firm sponge has the same flammability characteristics as the 1062 soft sponge. It too is self-extinguishing above a thickness of 3/8 inch if the skin is removed. An example of these flammability characteristics is illustrated in figure 4. Mosites 1062-C sponge is a good replacement for polyurethane helmet liners, seat cushions, and crew couches.

Mosites 1061-D, -K, and -J Fluorels and 1067 and 1067-B Fluorels.- This series was developed by Mosites to improve the physical properties and provide a range of materials for special fabrication applications.

The 1061-D dense sheet has these physical properties:

Durometer, Shore A	68
Tensile strength, psi	1486

Elongation, percent 150

ASTM tear strength (Die C), lb/in. 106

Compression set, percent

After 22 hr at 350° F 19.4

Specific gravity 2.02

Physical properties of the 1061-K dense sheet are the following:

Durometer, Shore A 75

Tensile strength, psi 1816

Elongation, percent 125

ASTM tear strength (Die C), lb/in. 129

Compression set, percent

After 22 hr at 350° F 19.5

Specific gravity 2.02

Mosites 1061-J dense sheet exhibits the following physical properties:

Durometer, Shore A 78

Tensile strength, psi 2029

Elongation, percent 125

ASTM tear strength (Die C), lb/in. 128

Compression set, percent

After 22 hr at 350° F 26.6

Specific gravity 2.02

Mosites 1067 dense sheet has these physical characteristics:

Durometer, Shore A 83

Tensile strength, psi 2500

Elongation, percent	200
Modulus, 100 percent	1789
ASTM tear strength (Die C), lb/in.	172
Compression set, percent	
After 22 hr at 350° F	14.9
Shrinkage, percent	
Length	4.4
Width	2.9
Specific gravity	2.12

Physical properties of Mosites 1067-B dense sheet are:

Durometer, Shore A	81
Tensile strength, psi	2526
Elongation, percent	175
Modulus, 100 percent	1644
ASTM tear strength (Die C), lb/in.	171
Compression set, percent	
After 22 hr at 350° F	14.7
Shrinkage, percent	
Length	4.4
Width	2.5
Specific gravity	2.03

This series of Fluorel dense sheet stock, 1/16 to 1/8 inch thick, requires a 24-hour cure at 400° F. After cure, they are nonflammable under conditions of bottom ignition, 16.5 psia in 100 percent oxygen.

Mosites 1066 Fluorel adhesive.—Mosites has developed this adhesive compound from their basic 1059 formulation. This adhesive has shown excellent application in bonding spacecraft components although it may

be necessary to first prime the surface. Use of this adhesive imparts nonflammable characteristics to the total lamination, the extent of the nonflammability dependent, of course, on the substrate.

Under 16.5 psia in 100 percent oxygen, a sample of Telfon Velcro pile (P490) adhered to aluminum with the 1066 adhesive did not ignite. During the flame impingement test, the sample charred and produced smoke in the area of heat, but the remainder of the sample did not burn. The test was conducted for 4.5 minutes, while the opposite side of the aluminum reached equilibrium temperature of 580° F in 20 seconds.

Samples of both uncoated and Fluorel 1059-coated Beta adhered with the 1066 adhesive were tested for flammability in 100 percent oxygen, at 16.5 psia with bottom ignition. The coated sample burned with copious quantities of smoke at 0.29 in/sec. The uncoated sample burned at a rate of 0.071 in/sec which was reduced to 0.039 in/sec when the adhesive was used without primers. These results can be compared to rates of 1.66 in/sec for Neoprene N136B adhesives.

This adhesive has been successfully used for laminating Fluorel foam to Lexan without application of heat. Also, samples of coated and uncoated Beta cloth have been bonded together with this adhesive and tested for shear and tear strengths. In each instance, the material failed before the bond sheared (fig. 5).

Raybestos-Manhattan Nonflammable Materials

Raybestos-Manhattan L-3203-6 Fluorel. - This mill sheet is of Raybestos-Manhattan formulation, utilizing the basic 3M Company copolymer. With the ingredients added by Raybestos-Manhattan, this material and its variations possess the best nonflammability characteristics of all the Fluorels developed to date. No cure is required for nonflammability properties, although curing does enhance the physical characteristics. Actually, the degree of cure determines to a great extent the properties of this compound, necessitating strict adherence to the cure cycles specified for a particular fabrication.

Physical properties of L-3203-6 are contained in the following data:

Original properties

Durometer, Shore A

Press cure, 30 min at 320° F	82
Postcure, 16 hr at 250° F	96
Postcure, 16 hr at 300° F	95
Postcure, 16 hr at 350° F	95
Postcure, 16 hr at 400° F	97

Tensile strength, psi

Press cure, 30 min at 320° F	557
Postcure, 16 hr at 250° F	1660
Postcure, 16 hr at 300° F	1548
Postcure, 16 hr at 350° F	1500
Postcure, 16 hr at 400° F	1558

Elongation, percent

Press cure, 30 min at 320° F	362
Postcure, 16 hr at 250° F	113
Postcure, 16 hr at 300° F	88
Postcure, 16 hr at 350° F	75
Postcure, 16 hr at 400° F	75

ASTM tear strength (Die C), lb/in.

Postcure, 16 hr at 350° F	128
-------------------------------------	-----

Specific gravity	2.11
----------------------------	------

Mooney at 212° F	147
----------------------------	-----

Air-oven aged 7 days at 400° F

Tensile strength, psi	1155
Durometer, Shore A	98
Elongation, percent	50

Bashore resilience, percent	5
---------------------------------------	---

Compression set, percent

After 22 hr at 400° F	64.5
---------------------------------	------

Low temperature (Gehman flexibility), °F

Low temperature (Brittle point), °F

Electrical properties

Frequency

Dielectric constant at 25° C

Dissipation factor at 25° C

Thermal conductivity, Btu/ft.²/°F/in. 1.37

At 16.5 psia, samples under 0.030-inch thickness will be destroyed. Beyond this dimension, the material is nonflammable by Apollo standards. At 6.2 psia, there is no thickness limitation. This material has also passed the liquid oxygen (lox) impact compatibility test at the Marshall Space Flight Center, Huntsville, Alabama.

The L-3202-6 compound has been successfully used for coating Beta, and for fabricating electrical harness boots, belts, harnesses, flight boot soles, and bladder material (fig. 6).

This formulation has also been developed as a nonflammable adhesive. Samples of Beta cloth were bonded together with this adhesive, with and without primers. In all instances, the fabric failed before the bond broke.

Raybestos-Manhattan adhesive L-3203-6 with and without primers is nonflammable in 100 percent oxygen at 16.5 psia with bottom ignition. Raybestos-Manhattan is continuing study on this compound to improve its extrusion characteristics.

Raybestos L-3236 Fluorel.- This Fluorel-silicone-rubber compound is supplied in mill sheet form. This material has extremely good cold temperature properties although its tensile strength could be improved. The L-3236 has the following physical characteristics:

Original properties

Durometer, Shore A	86
Tensile strength, psi	320
Elongation, percent	50
Specific gravity	1.85

Air-oven aged 7 days at 400° F

Durometer, Shore A	+3 (89)
Tensile strength, psi	+2.5 (328)
Elongation, percent	0 (50)

Bashore resilience, percent 10

Compression set, percent

Low temperature (Gehman flexibility), °F

Low temperature (Brittle point), °F

Electrical properties

Volume resistivity, ohm/cm	9.6×10^{12}
Dielectric strength, V/mil	525

Frequency

Dielectric constant at 25° C

100 Hz	6.90
1 kc	6.66
10 kc	6.45
100 kc	6.27

Dissipation factor at 25° C

100 Hz	0.0167
1 kc	0.0202
10 kc	0.0164
100 kc	0.0123

Thermal conductivity, Btu/ft²/°F/in.

1.21

A sample of 0.060-inch-thick L-3236 sheet, cured for 18 hours at 350° F, was tested for nonflammability at 16.5 psia, bottom ignition, oxygen atmosphere, and found to be self-extinguishing in 13 seconds. The ignition wire was reactivated for 30 seconds and the sample did not ignite. Again, at 16.5 psia, samples should not be used below 0.060 inch.

This compound has been fabricated into tubing requiring good low-temperature properties. Due to the low tensile strength, it is recommended that the tubing be reinforced.

Raybestos L-3251-3 Fluorel.- This mill sheet is a derivative of the L-3203-6 compound to obtain improved extrusion properties. Although this material does require curing at 350° F for 16 hours for nonflammability, burning characteristics are superior to the L-2231 compound.

The L-3251-3 mill sheet has the following physical characteristics:

Press cure 30 min at 320° F

Postcure 16 hr at 350° F

Original properties

Durometer, Shore A

Press cure, 30 min at 320° F	80
Postcure, 16 hr at 350° F	96

Tensile strength, psi

Press cure, 30 min at 320° F	500
Postcure, 16 hr at 350° F	1820

Elongation, percent

Press cure, 30 min at 320° F	325
Postcure, 16 hr at 350° F	75

ASTM tear strength (Die C), lb/in.

Postcure, 16 hr at 350° F	61.5
Specific gravity	2.08

Mooney at 212° F	120
Air-oven aged 7 days at 400° F	

Durometer, Shore A	+1 (97)
Tensile strength, psi	-1.7 (1787)

Elongation, percent	-33.3 (50)
Bashore resilience, percent	4

Compression set, percent

After 22 hr at 400° F	71.9
Low temperature (Gehman flexibility), °F	

T2	+30
T5	+12

T10	+5
T100	-11

T100	-11
Low temperature (Brittle point), °F	

1/16 gage	+8.6° F
1/32 gage	-9.4° F

1/64 gage	-43.6° F

Electrical properties

Volume resistivity, ohm/cm	2.9×10^{13}
Dielectric strength, V/mil	538

Frequency

Dielectric constant at 25° C

100 Hz	7.20
1 kc	7.03
10 kc	6.76
100 kc	6.23

Dissipation factor at 25° C

100 Hz	0.0073
1 kc	0.0214
10 kc	0.0350
100 kc	0.0483

Thermal conductivity, Btu/ft²/°F/in.

1.34

During flammability testing, a sample of 0.015-inch sheet stock did not ignite with bottom ignition at 6.2 psia, 100-percent oxygen atmosphere. The ignition wire was reactivated for 30 seconds and the sample burned with a smoky yellow flame at 0.063 in/sec, 16.5 psia. A 0.040-inch sample did not ignite, even after the wire was reactivated.

Because of its superior flammability characteristics, this compound is under investigation as a fireproof coating. It has also been successfully evaluated for extruding hoses and tubing for the Apollo spacecraft. The L-3251-3 Fluorel stock has also been evaluated for coating gloves and for the manufacture of flight boot soles (fig. 7).

Raybestos-Manhattan L-3322-2 and L-3322-5 Fluorels.— These Fluorel dense sheets were formulated to modify the L-3203-6 compound for improved extrusion qualities and flexibility. Both the L-3322-2 and L-3322-5 compounds are nonflammable under spacecraft conditions of 16.5 psia, bottom ignition, 100-percent oxygen atmosphere. L-3322-2 Fluorel has the following physical properties:

Press cure	30 min at 320° F
Postcure	16 hr at 350° F

Durometer, Shore A	95
Tensile strength, psi	999
Elongation, percent	125

L-3322-5 Fluorel exhibits the following physical characteristics:

Press cure	30 min at 320° F
Postcure	16 hr at 350° F
Durometer, Shore A	84
Tensile strength, psi	1455
Elongation, percent	175

These Fluorels have been evaluated for the fabrication of hoses and boot soles. They have also been used for coating Beta fabric for nonflammability and flexibility.

Raybestos-Manhattan Fabricated Fluorel Products

Raybestos-Manhattan RL-3557 tubing.- This designation has been given to Raybestos-Manhattan extruded tubing made from their L-3251-3 compound (fig. 8). Initial laboratory samples were more porous than desirable; however, it is believed that porosity can be improved by variation in the curing procedure.

Raybestos-Manhattan RL-3529 harness belt.- This number refers to a lifevest harness made from the L-3203-6 compound. At 0.050 inch minimum, these belts are nonflammable under the rigid spacecraft conditions.

Raybestos-Manhattan RL-3492 compound.- This compound is a solution of L-3203-6 with 50 percent asbestos. The resulting paste does not need curing but the solvent must be driven off for nonflammability. This requirement for drying may result in some shrinking and applications should be chosen with care. However, the RL-3492 paste is extremely nonflammable and exhibits excellent barrier properties.

A sample of the compound, cured under atmospheric conditions, did not ignite at 16.5 psia, 100-percent oxygen atmosphere. There was no ignition when the wire was reactivated for 40 seconds. A sample cured for 3 hours at 160° F did not burn on first ignition or on reactivation of the wire for 1 minute, 10 seconds.

This material has been successfully evaluated as a nonflammable caulking compound and conformal coating as illustrated in figure 9. However, the 50-percent asbestos compound alone has not been able to pass humidity tests. Circuit breakers, coated with Fluorel RL-3492, are presently under investigation for flammability and humidity retention. An additional sample of breakers will be coated with a solution of L-3203-6, which will deposit a rubber coating on the breakers before overcoating with RL-3492. It is hoped this technique will obtain superior humidity resistance.

In tests at North American, RL-3492 was brush coated (five coats totaling 0.045 inch with 1/2-hour air dry between coats and 5 hours oven drying at 150° F) on a circuit breaker. This coating technique successfully prevented propagation of a silicone undercoat fire under spacecraft conditions. This double-coated circuit breaker has also passed North American humidity tests.

Raybestos-Manhattan RL-3550 compound.— This compound is a solution of L-3203-6 with 25 percent asbestos. Flammability properties and applications conform with the RL-3492 compound, but the RL-3550 formulation is softer, more flexible and has improved humidity properties. The RL-3550 compound when postcured at 300° F for 16 hours, has the following physical properties:

Tensile strength, psi	100
Elongation, percent	25
Solids, percent	50
Specific gravity, wet	1.20
Specific gravity, dry	1.94

Raybestos-Manhattan RL-3550 can be poured into desired areas, or can be applied with a spatula. Heavy coatings should be allowed to dry 24 to 48 hours. If a less viscous material is needed or desired, methyl ethyl ketone solvent can be used to reduce solids.

Raybestos-Manhattan RL-3550 compound has been successfully sprayed as a conformal coating on an electrical circuit breaker. The RL-3550 formula has also been coated on RTV silicone rubber and has extinguished ignition of the silicone.

The RL-3550 material, coated 1/8 to 3/16 inch thick on RayChem coaxial cable (fig. 10), has successfully extinguished fire under spacecraft conditions at 6.2 and 16.5 psia. Lunar module circuit breakers were coated with approximately 1/8-inch Fluorel RL-3550 (fig. 11). An uncoated breaker was placed 1/2-inch between the coated breakers and ignited in 100 percent oxygen at 16.5 psia with bottom ignition. The ignited breaker burned to completion and did not ignite the adjacent coated lunar module breakers. The oxygen content was tested and found to be 80 percent in the chamber at the end of the combustion.

When using this compound for conformal coatings, it is important to obtain maximum drying at atmospheric conditions to avoid bubbling and cavities. There is also more skinning effect on drying with the lower asbestos content.

Raybestos-Manhattan RL-3494-1, 3494-2, and 3494-3 Fluorel-coated Beta. - These numbers refer to Fluorel coated on heat-cleaned Beta 4190X. Pickup is 17.8, 22.6, and 25 percent, respectively. All Fluorel-coated Beta samples are self-extinguishing in 3 seconds at 16.5 psia, bottom ignition, in a 100-percent oxygen atmosphere. Production samples of 22-percent pickup Fluorel-coated Beta have been ordered for further evaluation as space suit material and for use as exhaust duct covering.

Raybestos-Manhattan RL-3587 white Fluorel-coated Beta. - Raybestos-Manhattan has coated Beta with white Fluorel L-3203-6 which has been cured for 16 hours at 200° F, with 22.4-percent pickup. The superior flammability of this material is illustrated in figure 12.

FABRICATION OF NONFLAMMABLE APOLLO SPACECRAFT COMPONENTS AND RELATED EQUIPMENT

Hoses

R. E. Darling has successfully fabricated a Fluorel umbilical hose using the L-2231, 1059 or L-3217-1 formulations. This hose incorporates a stainless steel spiral and mesh, replacing the nylon reinforcement in present hoses. This hose was nonflammable according to Category A, MSC-A-D-66-3, Revision A.1.

The manufacturer is now in the process of fabricating an Apollo ship-set of umbilical hoses replacing the stainless steel mesh reinforcement with Nomex to obtain more flexibility. These hoses will be evaluated as a possible replacement for flammable silicone rubber hoses.

Using the same Fluorel compounds, R. E. Darling is fabricating flexible portable life support system (PLSS) hoses to and from the astronaut. Also, by utilizing a Nomex outer cover and reinforcement, they have fabricated a nonflammable Fluorel lock observer oxygen hose sample capable of withstanding 100 psig.

R. E. Darling has coated a silicone umbilical hose with Fluorel L-2231 which was cured for 24 hours at 320° F. The Fluorel successfully fireproofed the silicone hose and passed the Category A flammability tests. This company has also coated a 10-foot cobra cable with Fluorel L-2231. A 1-foot sample of this coated cable was received at MSC and successfully passed the flammability tests.

Sierra Engineering has fabricated nonflammable flexible oxygen-mask hoses using the Fluorel formulation. Figure 13 illustrates the nonflammability of this hose as compared with silicone hose under identical testing conditions. Fluorel has also been suggested as a possible material for manufacturing Apollo emergency pack hoses.

R. E. Darling has a development program underway to laminate silicone hoses with Fluorels L-3203-6, L-3251-3, L-3322-2, and L-3322-5, because of their superior nonflammability characteristics as compared to other Fluorels. These same Fluorels will also be evaluated for use as hoses and tubing.

Tubing

R. E. Darling has made tubing of the following dimensions from the L-2231 (1059, L-3217-1) formulation:

1/16-inch i.d. by 1/8-inch o.d.

3/16-inch i.d. by 5/16-inch o.d.

3/8-inch i.d. by 1/2-inch o.d.

1/4-inch i.d. by 7/16-inch o.d.

This nonflammable tubing (fig. 14) is used for the Apollo liquid cool garment.

Comformable Coatings

Raybestos-Manhattan RL-3550 compound, coated 1/8 to 3/16 inch thick on RayChem coaxial cable, has successfully extinguished the burning cable at 16.5 psia, 100-percent oxygen atmosphere. This coated cable is shown in figure 15.

Both Raybestos-Manhattan RL-3492 and RL-3550 have been evaluated for coating electrical components and have extinguished fires under space-craft conditions. Testing indicates that, by spraying the 25-percent solution on circuit breakers, fires resulting from internal shorts can be prevented or extinguished.

Foams

Mosites has successfully handmade a helmet liner (fig. 16) from 1062-C and has evaluated this material for use in camera packaging.

Sierra Engineering has adapted Mosites 1062 foam for earpieces and a newly designed helmet. Grumman Aircraft is presently evaluating Mosites 1062 for fabricating antenna shock absorbers.

Molded Products

Boot soles have been successfully fabricated by International Latex Corporation from the 3M Company's L-2231 and L-2248 and from Raybestos-Manhattan's L-3203-6 and L-3251-3 Fluorels. To date, International Latex prefers the L-3251-3 for physical and processing characteristics.

The 3M Company has manufactured a Fluorel magnet (fig. 17). A cured and an uncured magnet have been received and have passed the Category A flammability tests. B. F. Goodrich is also experimenting with Fluorel for manufacturing windowshade magnets.

Using the L-2231 and L-3203-6 Fluorels, CiCoil Company is successfully manufacturing an electrical harness boot for the biomedical harness (fig. 18).

A. C. Electronics has successfully evaluated 1059 for use as an optic shroud.

Coated Fabrics and Webbing

Abrasion testing has been conducted on Beta, coated with Raybestos-Manhattan RL-3494-1, 2 and 3. The results of this testing are presented in table II.

Fabric Research Laboratories have prepared Beta, PBI, and a combination of Beta and PBI fabric coated with Fluorel L-3203-6. All the coated Beta samples at room temperature cure using triethylene tetramine catalyst, cured at an elevated temperature or with no cure, were self-extinguishing at 6.2 and 16.5 psia in 100 percent oxygen with bottom ignition. The Fluorel-coated PBI and PBI and Beta combination samples at various cures did not prevent the PBI from burning.

Raybestos-Manhattan has coated Beta webbing with Fluorel L-3203-6 for use as suit harness and belting.

Adhesives

Mosites has successfully bonded Fluorel sponge 1062 to a Lexan helmet using their 1066 adhesive. This bond was obtained without heat using the following procedure:

- a. Brush on one coat of Mosites 1066 adhesive to the area on the polycarbonate to be bonded. Let air dry 1 hour.
- b. Brush on one coat of Mosites 1066 adhesive to area on the 1062 Fluorel sponges to be bonded. Let air dry 1 hour.
- c. Place adhesive-coated surfaces of the Mosites 1062 Fluorel sponge and the polycarbonate together.
- d. Assure intimate contact by applying 5 pounds of pressure, by hand, to the area being bonded.

Samples of Beta cloth were adhered with Mosites 1066 adhesive, both with and without primers.

The shear strengths of the samples were determined on the Instron Tensile Tester in accordance with Federal Test Method Standard No. 175, Method 1041.1. The results, compared with N136B Neoprene adhesive, are given in table III.

To bond Beta cloth to itself using Mosites 1066 adhesive:

- a. Apply one coat of Mosites 1066 B-primer to area on each piece to be bonded. Let air dry for 30 minutes.

b. Apply one coat of Mosites 1066 C-primer to area on each piece to be bonded. Let air dry for 30 minutes.

c. Apply Mosites 1066 adhesive to primed area on each piece to obtain a 0.010-inch dry film. Let air dry 1 hour. This procedure will be repeated to obtain three coats; each additional coat should air dry for 15 minutes.

d. Lay up cloth with coated side together.

e. Cure 30 minutes at 330° F, 15 psi.

f. Postcure 24 hours at 400° F.

Raybestos-Manhattan adhesive L-3203-6 has been tested with and without primer on Beta. Shear and peel strengths, as determined on the Instron Tensile Tester, are given in table IV. These results are compared with N136B Neoprene adhesive, Beta samples.

The Fluorel compounds and their fabrications discussed in this report represent only a small portion of the total investigated under this program. They do, however, constitute those Fluorel elastomers considered most promising for replacing flammable spacecraft components.

MAJOR PARTICIPATING COMPANIES IN THE FLUOREL PROGRAM

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TABLE I.- APOLLO CERTIFICATION STATUS^a

Fluorels	Flammability	Total organics and CO, ppm			Odor			Flash and fire					
		Samples			Dilution with O ₂			Samples					
		1	2	3	8 hr	24 hr	48 hr	72 hr	1 to 29	1 to 9	0	1	2
Mosites 1066 adhesive	0.0	0.0	0.0										
Raybestos-Manhattan L-3203-6	.0	.0	.0	24.2	53.1	20.6	20.7	1	3	10			
Raybestos-Manhattan L-3235	.0	.0	.0										
Raybestos-Manhattan RL-3492	.0	.0	.0										
Raybestos-Manhattan L3251-3	.0	.0	.0										
The 3M Company L-2231	.0	.0	.0	.75					9	13	26	No fire	No fire
												No fire	No fire

^aAll values at 16.5 psia.

TABLE II.- FLUOREL-COATED BETA ABRASION TESTS

Fluorels	Pickup, percent	Results cycles
Stoll Wear tester inflated diaphragm 5 psig		
RL-3494-1	17.8	122
RL-3494-2	22.6	228
RL-3494-3	25.0	212
Taber abrader CS-17 wheels 500 g wt. on each wheel		
RL-3494-1	17.8	128
RL-3494-2	22.6	165
RL-3494-3	25.0	208
Wyco abrader 6 lb tension		
RL-3494-1	17.8	18
RL-3494-2	22.6	14
RL-3494-3	25.0	14

TABLE III.—MOSITES 1066 ADHESIVE — BOND STRENGTHS OF BETA LAMINATIONS

Lamination	Primer	Cure	Peel strength, lb/in.	Shear strength, lb/in.
Beta with 1066	No	24 hr at 400° F	6.50	120 — Beta failed
Beta with 1066	Yes	24 hr at 400° F		85 — Beta failed
N136B Neoprene			3.00	120 — Beta failed

TABLE IV.— RAYBESTOS-MANHATTAN L-3203 ADHESIVE — BOND STRENGTHS OF BETA LAMINATIONS

Lamination	Primer	Cure	Peel strength, lb/in.	Shear strength, lb/in.
Beta with L-3203-6	No	16 hr at 250° F	1.50	120 — Beta failed
Beta with L-3203-6	No	24 hr at 150° F	1.73	120 — Beta failed
Beta with L-3203-6	Yes	24 hr at 400° F	1.20	120 — Beta failed
Beta with L-3203-6	Yes (2)	24 hr at 400° F	.80	59 — Broke at bond
N136B Neoprene			3.00	120 — Beta failed

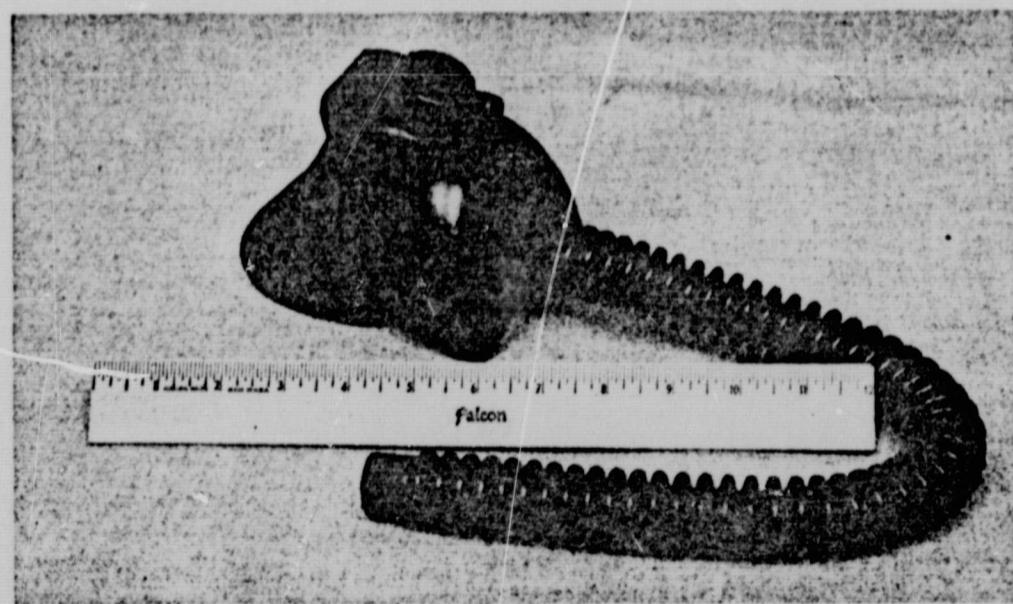
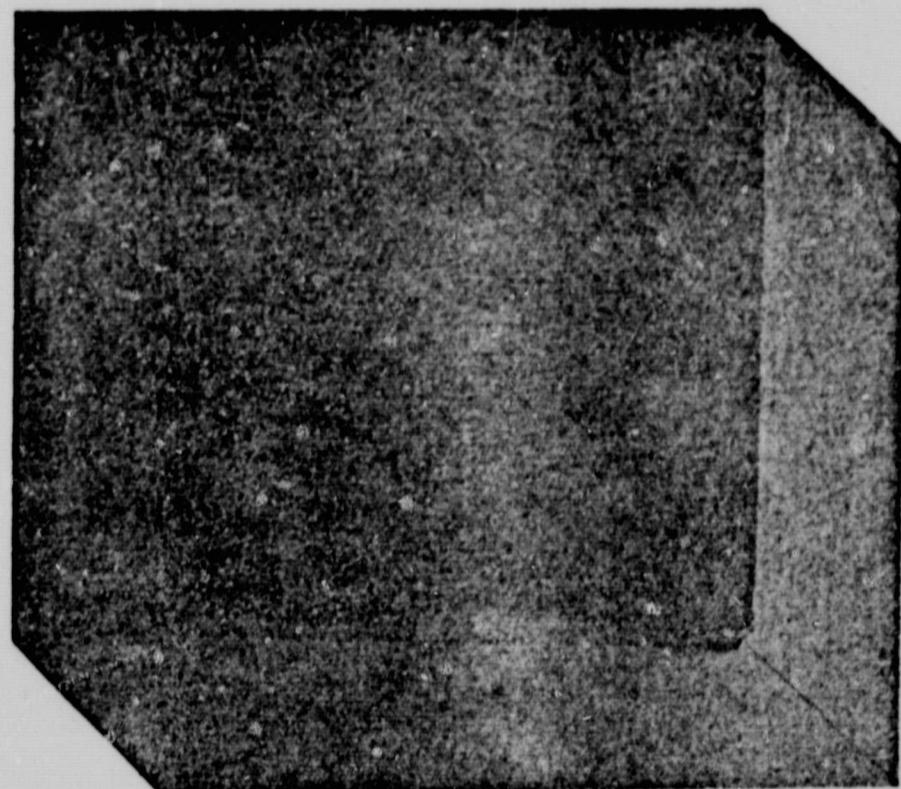


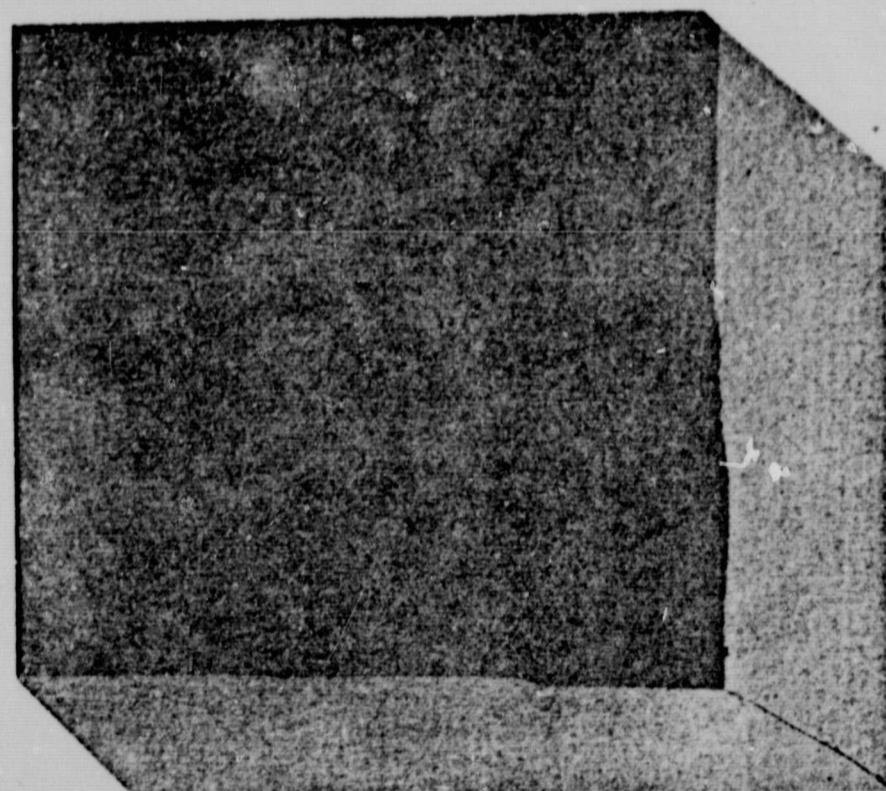
Figure 1.- Fluorel oxygen mask and hose, Sierra Manufacturing Company.



Figure 2.- Nonflammable Fluorel L-3217-1 flexible tubing,
R. E. Darling Company.

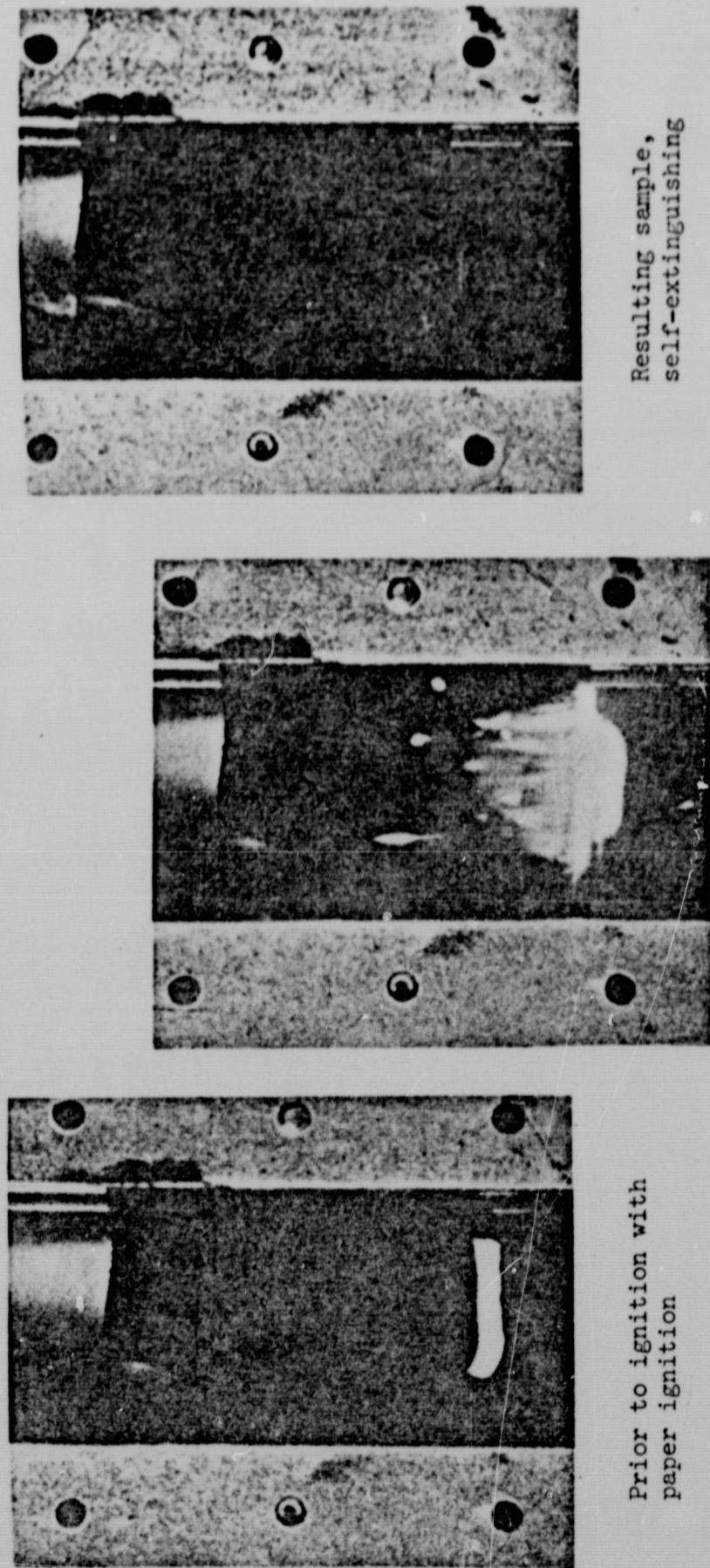


Mosites 1062 sponge
without skin



Mosites 1062 sponge
with skin

Figure 3.- Nonflammable Fluorel 1062 sponge, Mosites Rubber Company.



Prior to ignition with
paper ignition

During ignition

Resulting sample,
self-extinguishing

Figure 4.- Mosites 1062-C sponge flammability characteristics.

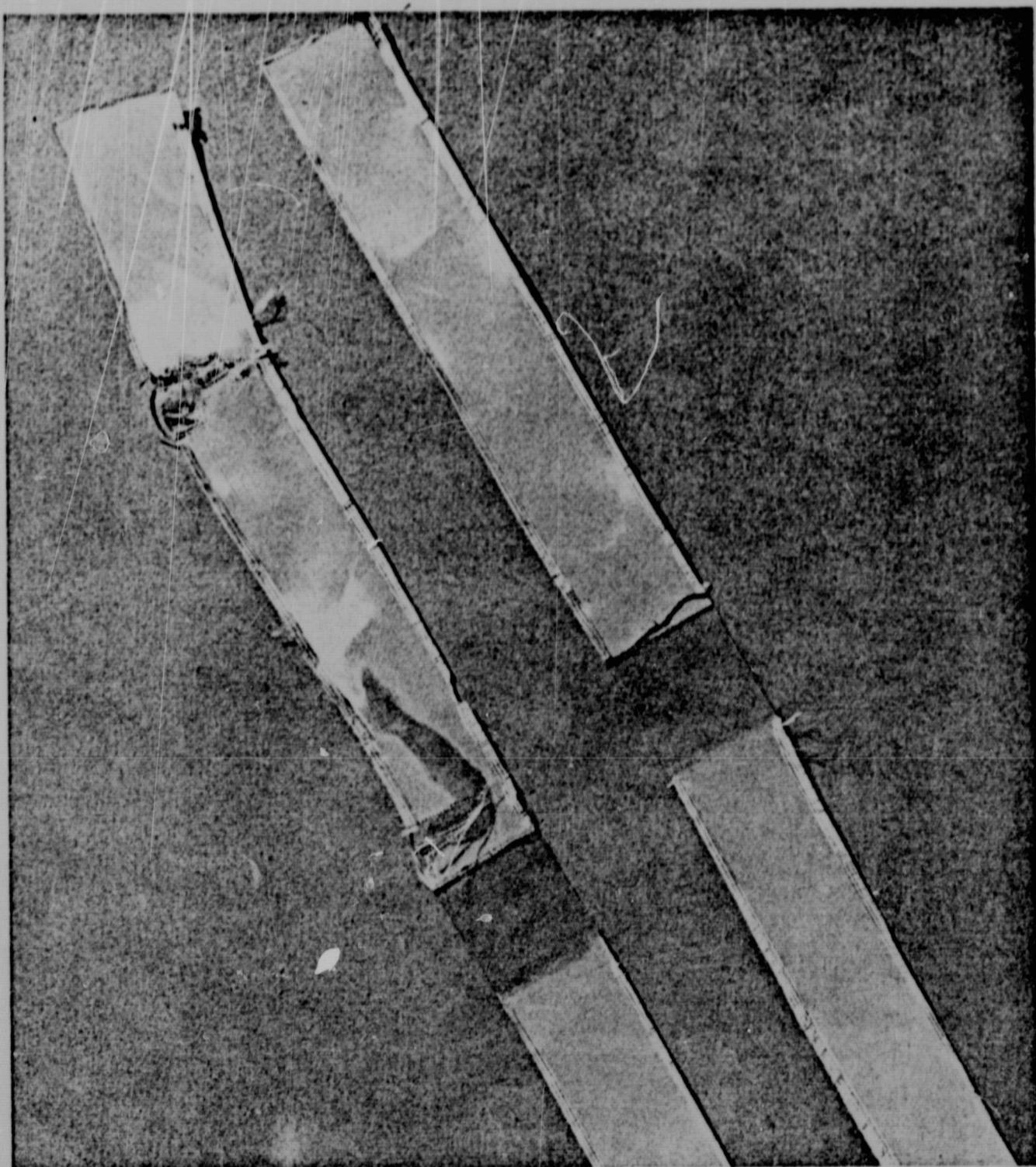


Figure 5.- Results from shear testing of Beta lamination. Fluorel 1066 adhesive manufactured by Mosites Rubber Company.

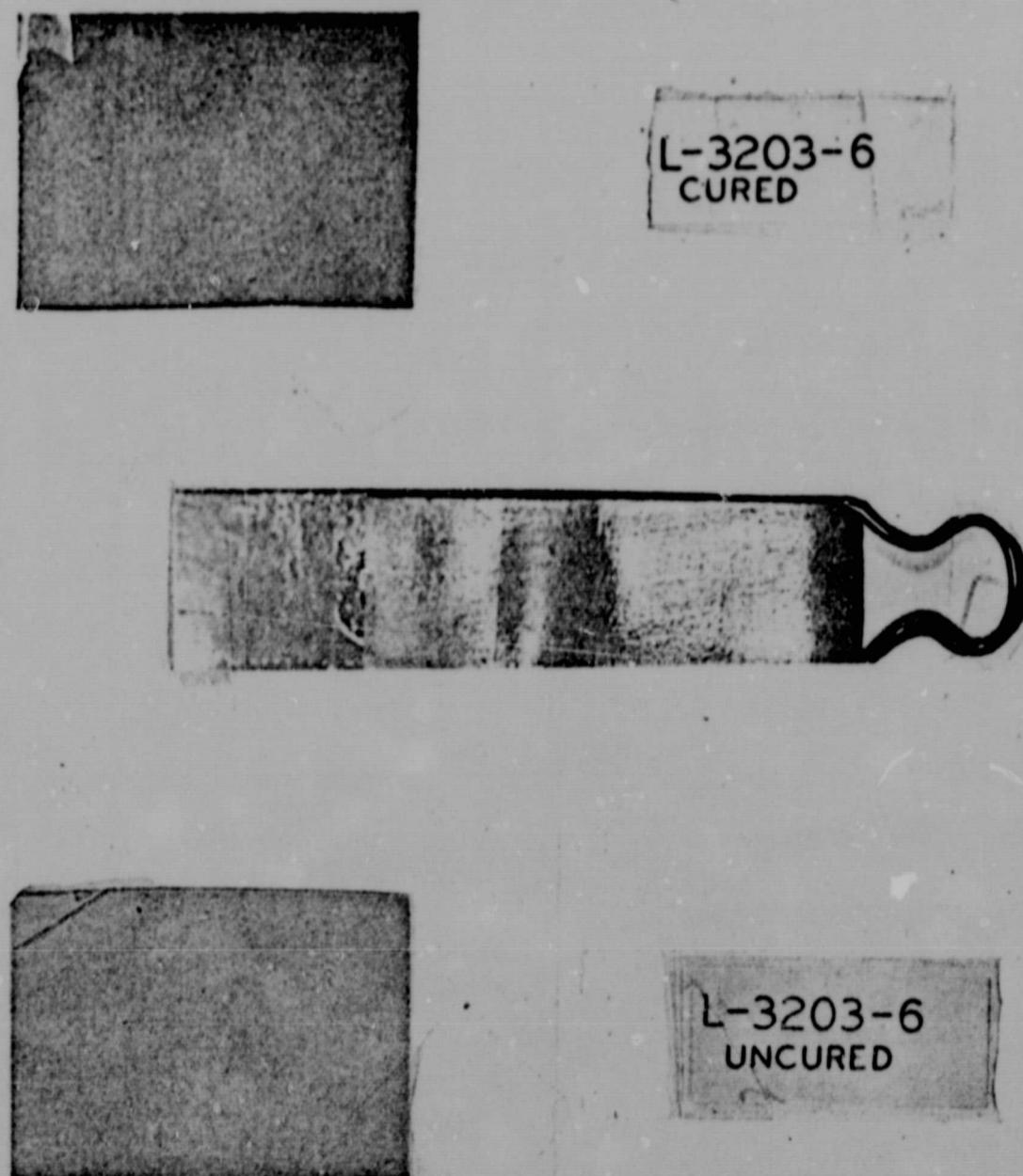


Figure 6.- Fluorel L-3203-6-coated belt with Beta webbing, Raybestos-Manhattan Company.

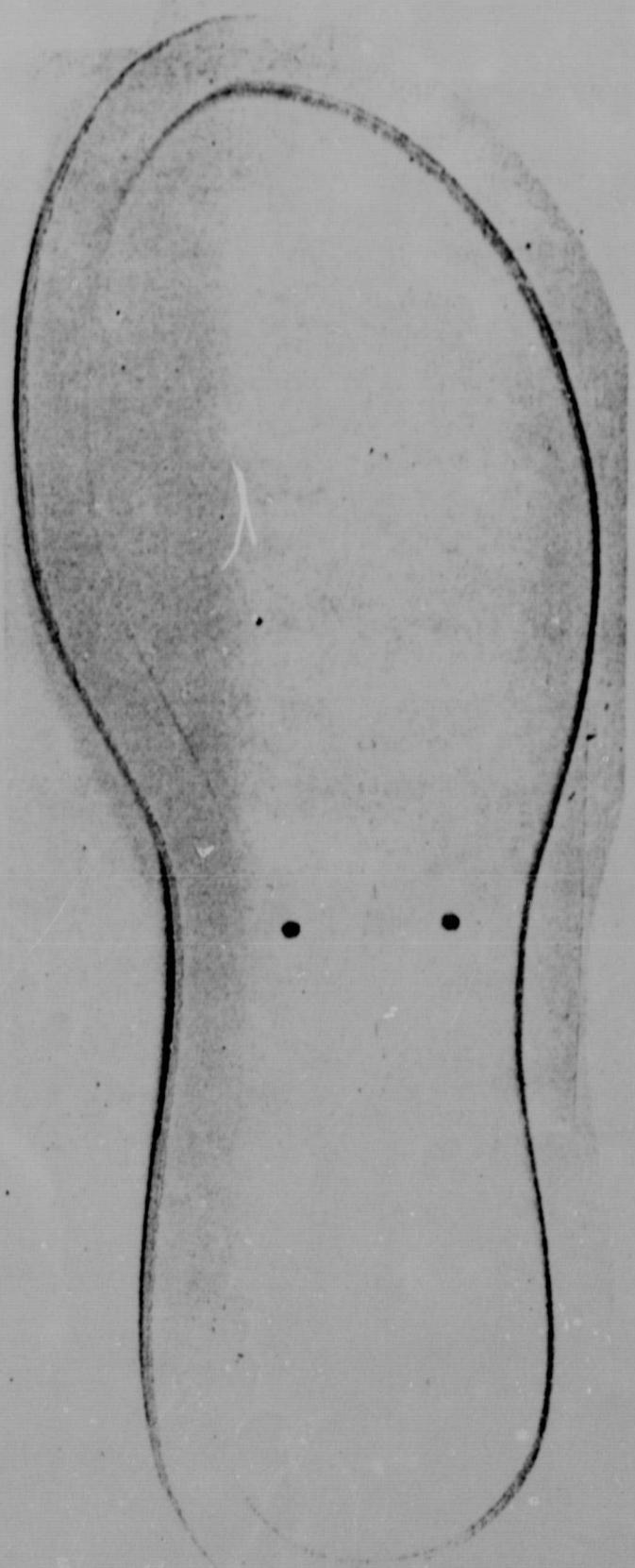


Figure 7.- Flight boot sole manufactured from L-3251-3 Fluorel, Raybestos-Manhattan Company.

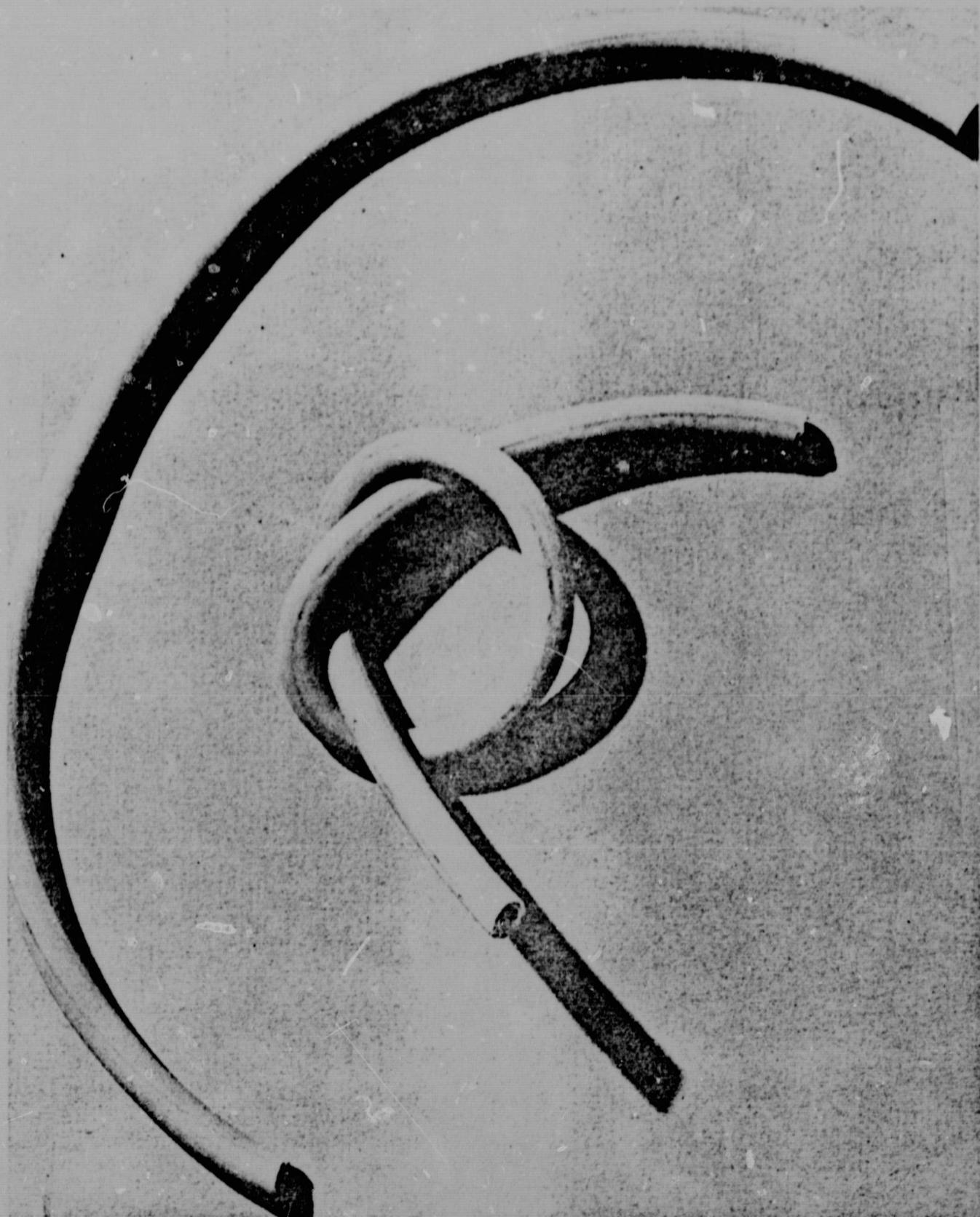


Figure 8.- Raybestos-Manhattan RL-3557 Fluorel flexible tubing.

NOT REPRODUCIBLE

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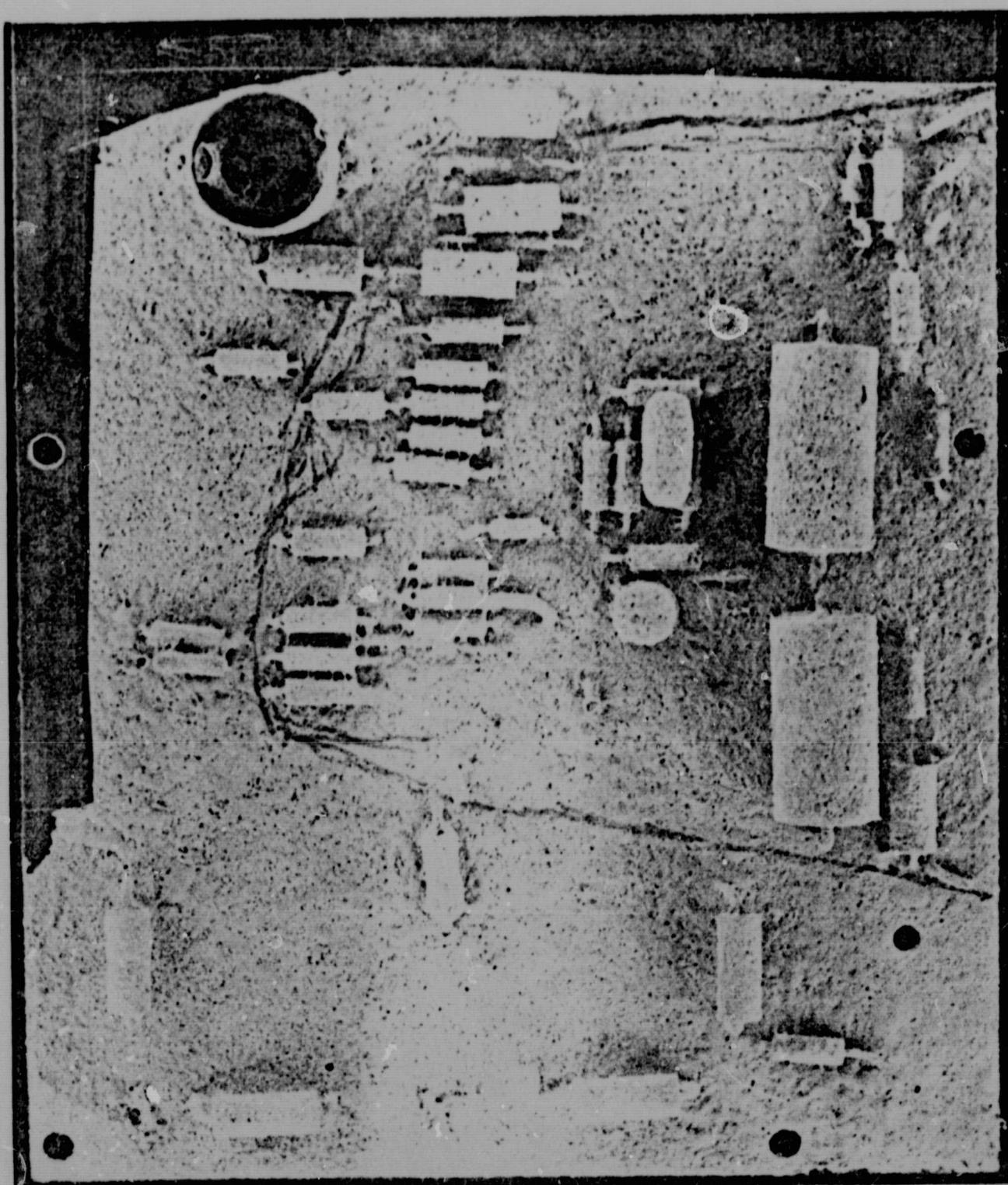


Figure 9.- Raybestos-Manhattan RL-3492 Fluorel conformable coating. Electrical panel prepared at the Manned Spacecraft Center.

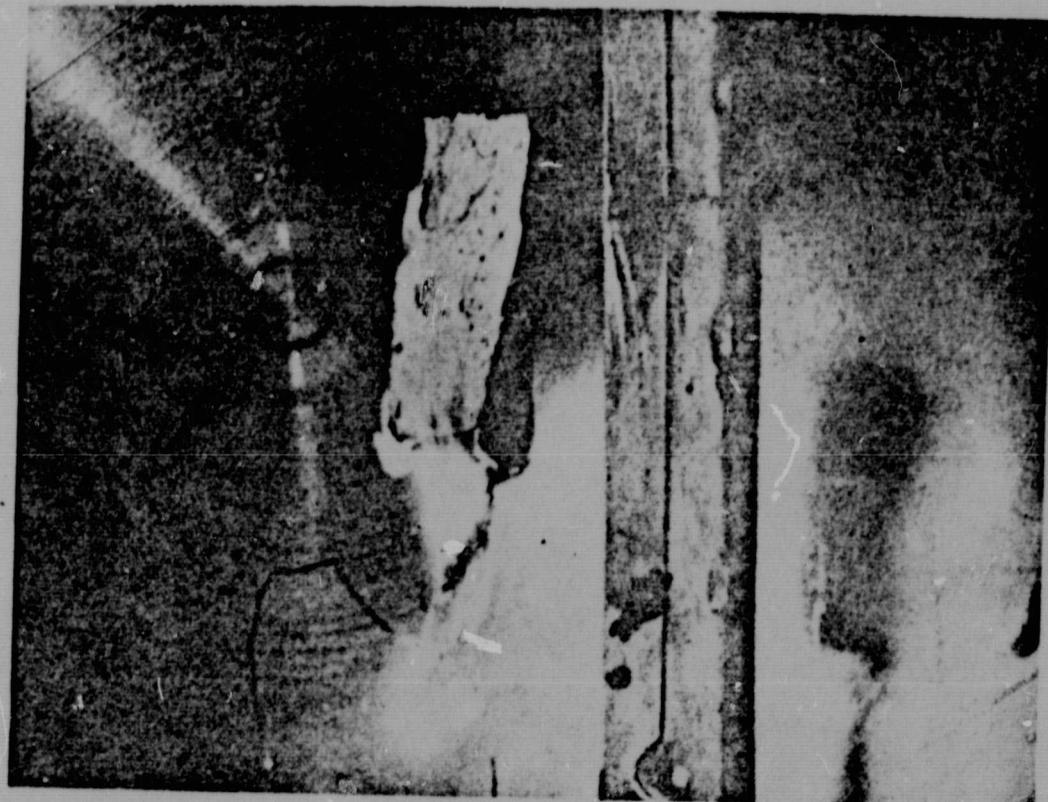
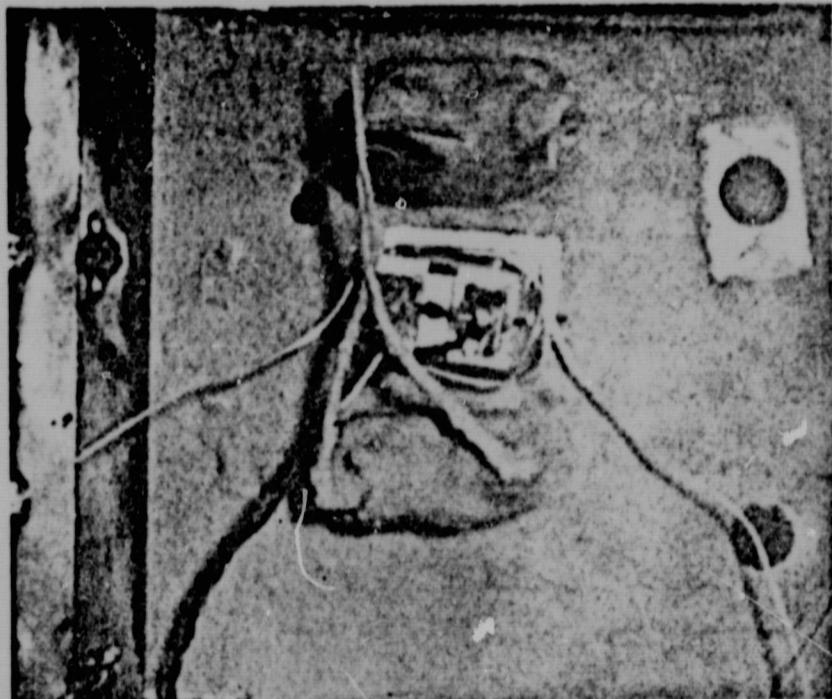


Figure 10.- Fluorel RL-3550 coating on RayChem coaxial cable,
Raybestos-Manhattan Company.

NOT REPRODUCIBLE

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Before ignition



After ignition

Figure 11.- Lunar module circuit breakers coated with Fluorel RL-3550. Samples prepared at the Manned Spacecraft Center.

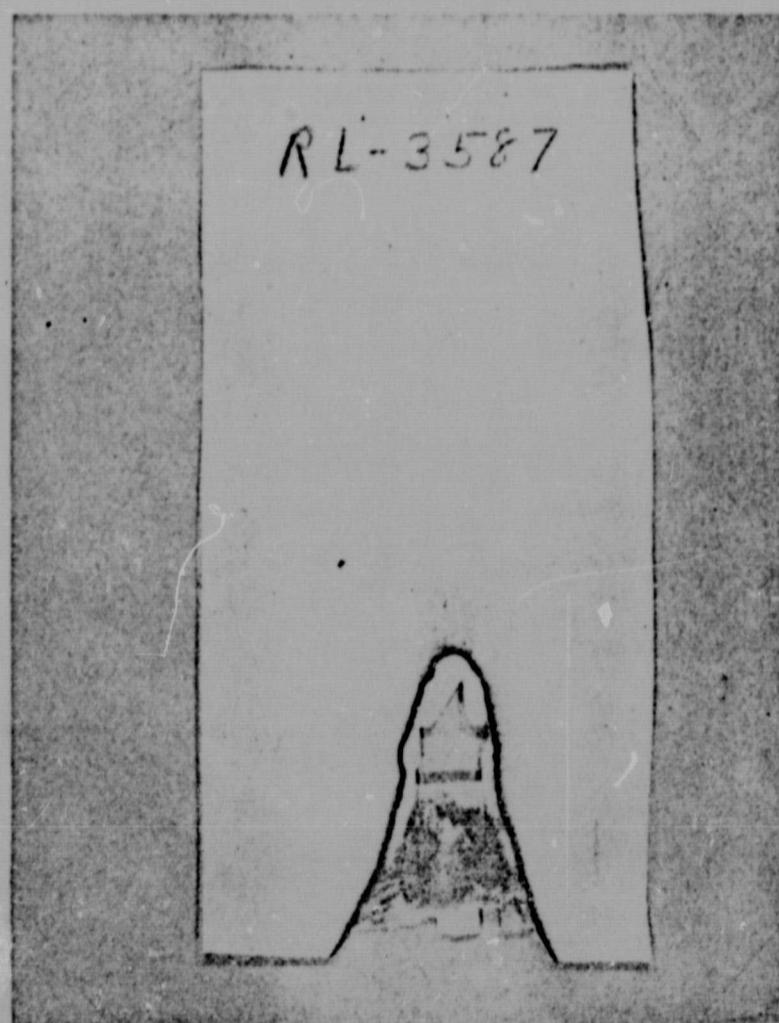


Figure 12.- White Fluorel RL-3587 Beta flammability test, Raybestos-Manhattan Company.

NOT REPRODUCIBLE

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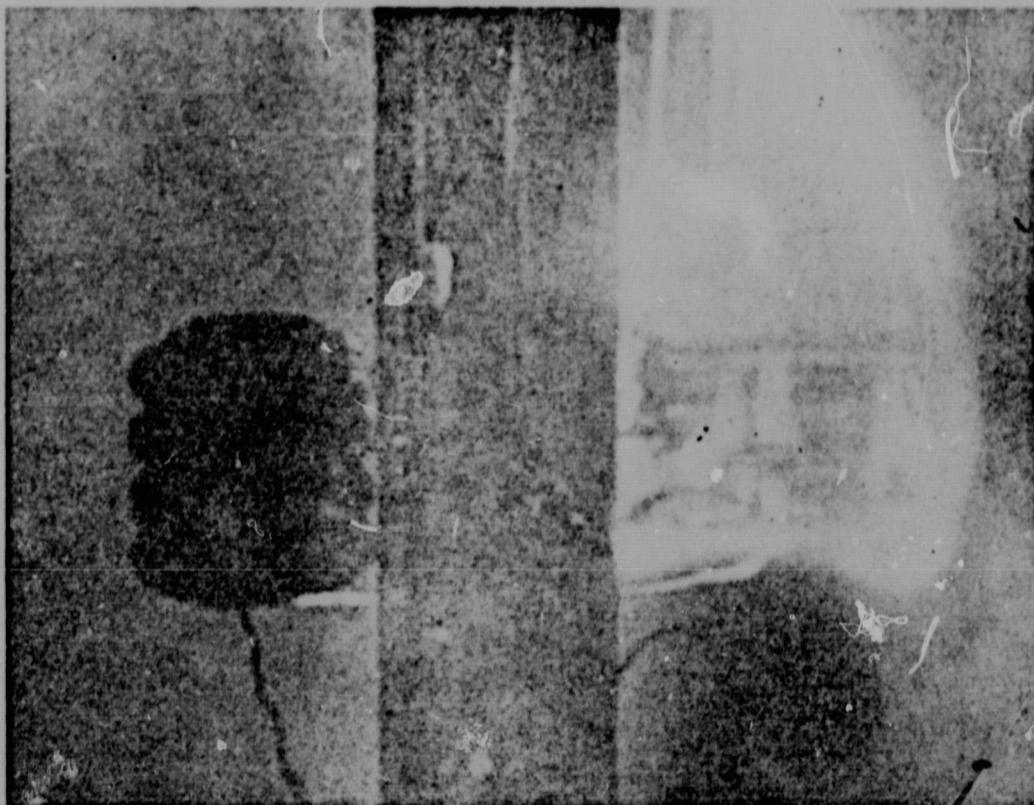


Figure 13.- Flammability comparisons between Fluorel and silicone oxygen hoses. Bottom ignition 100 percent oxygen, 16.5 psia.

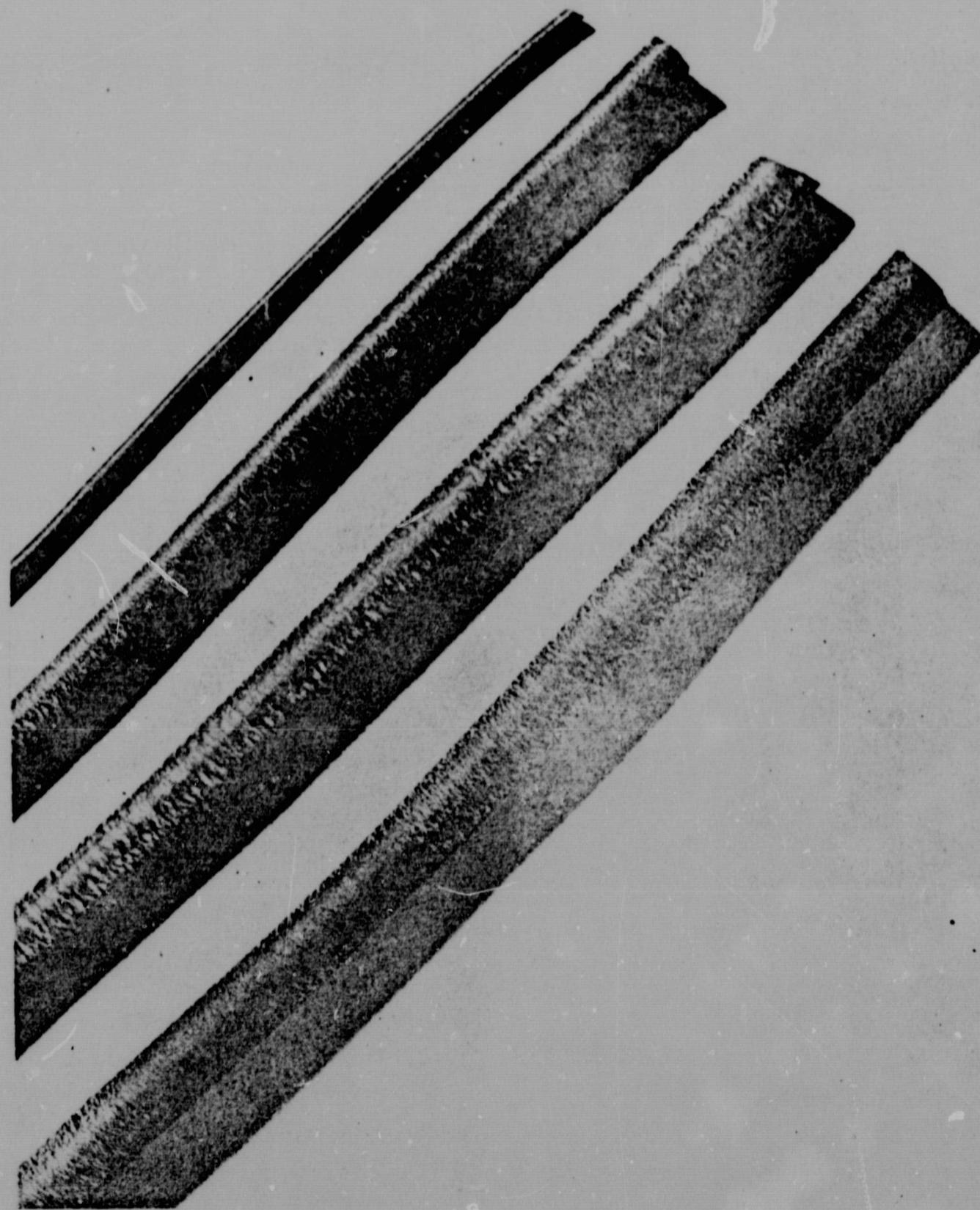


Figure 14.— Nonflammable Fluorel L-2251 tubing, R. E. Darling Company.

NOT REPRODUCIBLE

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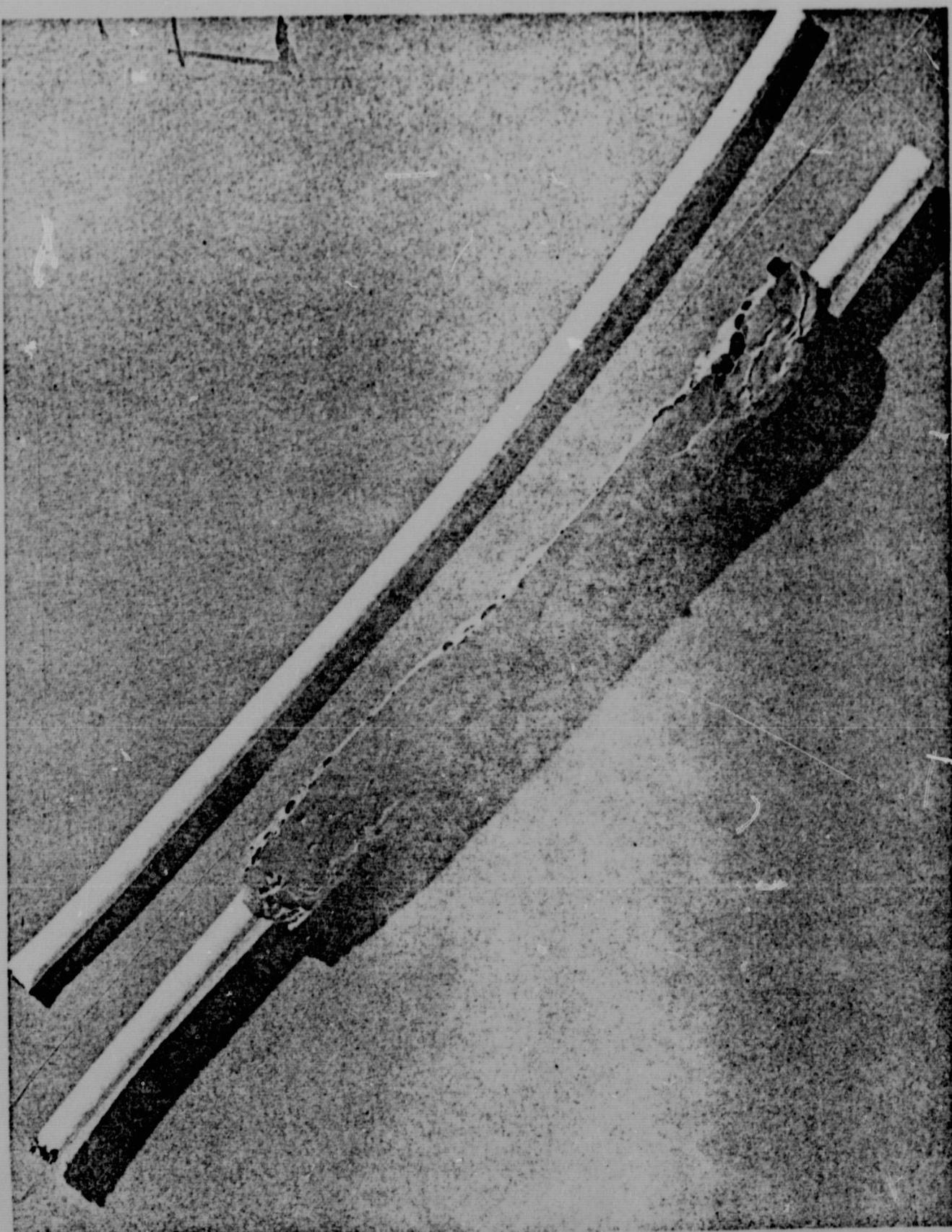


Figure 15.- Coaxial cable, coated with RL-3550 Fluorel asbestos compound,
Raybestos-Manhattan Company.

NOT REPRODUCIBLE

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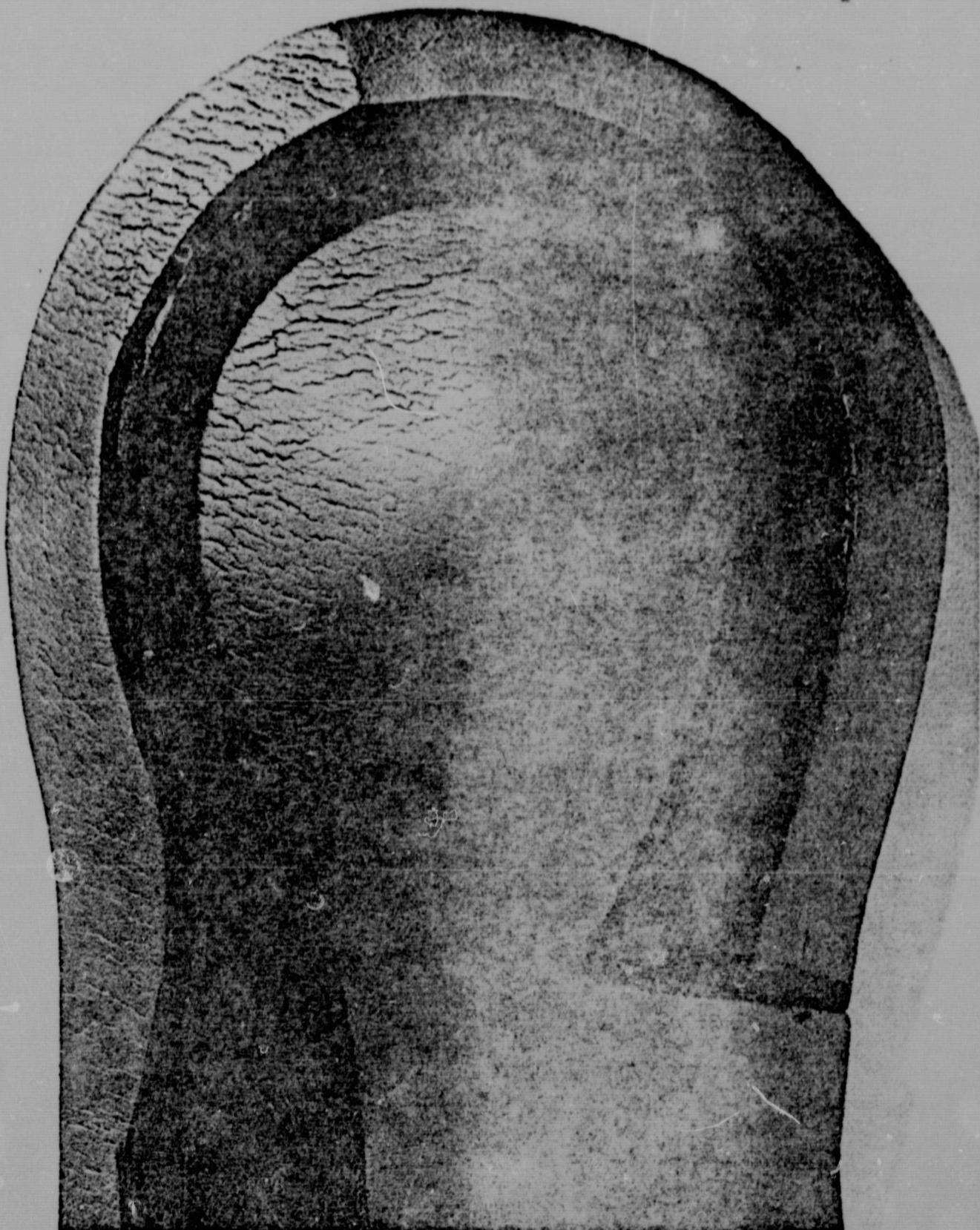


Figure 16.- Helmet liner fabricated from Mosites 1062-C foam,
Mosites Rubber Company.

NOT REPRODUCIBLE

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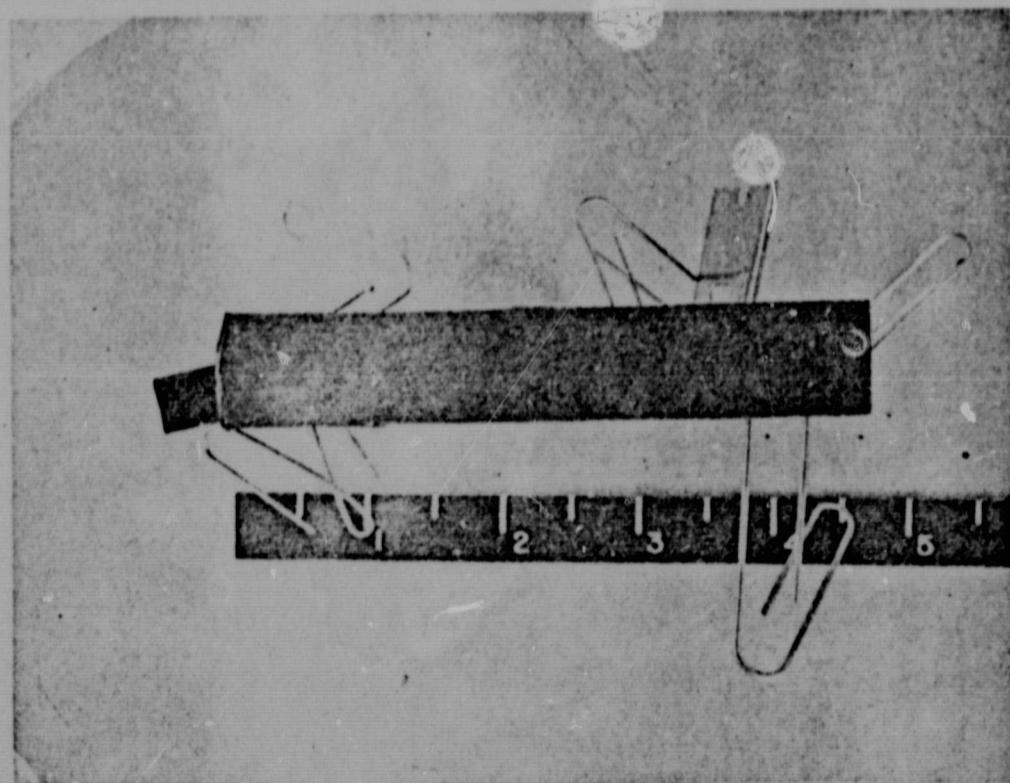
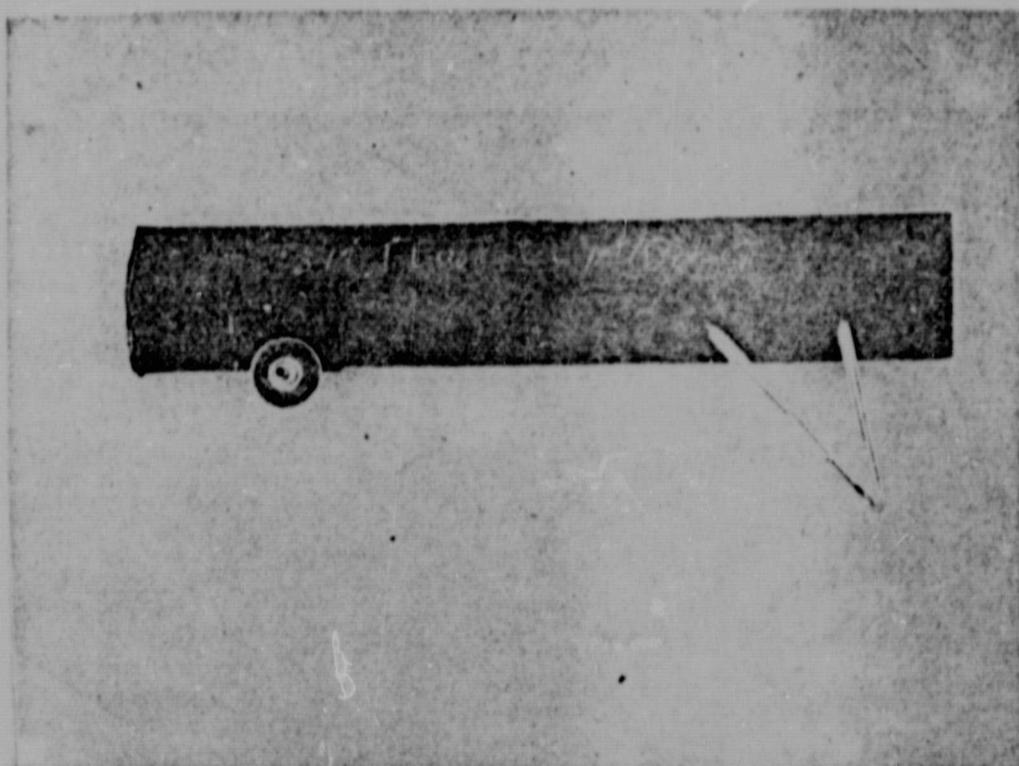


Figure 17.- Nonflammable Fluorel magnet, the 3M Company.

NOT REPRODUCIBLE

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Figure 18.- Fluorel electrical harness boot for biomedical harness,
CiCoil Company.